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IDENTIFICATION OF WEAR PARTICLES

Part I. Preliminary Investigations

LUBRICATION BRANCH
FUELS AND LUBRICATION DIVISION

MARCH 1978

TECHNICAL REPORT AFAPL-TR-78-14, Part I
Interim Report for Period 1 March – 30 September 1977

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AIR FORCE AERO PROPULSION LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
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This technical report has been reviewed and is approved for publication.

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A ferrographic reaction chamber has been constructed to permit the micro chemical identification of wear debris. Techniques including temperature coloring, chemical bluing, dissolution, pretreatment and crystal formation on metal particles have been investigated as potential useful methods for identification of wear debris on ferrograms. Temperature coloring, chemical bluing and crystal formation appear to be most promising of techniques studied, and will be pursued in subsequent effort.

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FOREWORD

This report describes an in-house effort conducted by personnel of the Lubrication Branch (SFL), Fuels and Lubrication Division (SF), Air Force Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio, under Work Unit 2303S301, "Wear Particle Identification." The effort was sponsored by the Air Force Office of Scientific Research, Washington D.C.

The work reported herein was performed during the period 1 March 1977 to 30 September 1977 with contributions by H. A. Smith, P. W. Centers, and W. R. Craig (AFAPL/SFL). The report was released by the authors in December 1977.

This report is an interim report on an effort which is continuing. Other reports will be published as phases of the work are completed.

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SECTION I

INTRODUCTION

1. Background

The analysis of used lubricating fluids for detecting abnormal wear conditions in machinery has been utilized for many years by various methods. The method or methods used for a specific system depends upon many factors including type lubricant used, type of equipment being monitored, conditions or environment under which the equipment is being used, critical nature of the equipment and cost-saving factors. Analysis techniques used include filtration testing, spectrometric oil analysis, and changes occurring in the physical and chemical properties of the lubricants such as viscosity, total acid or base number, electrical properties, etc.

Due to the critical and expensive nature of aircraft turbine engines, monitoring of used lubricants from these systems for diagnostic purposes is of major importance to both military and commercial applications of turbine engines. The importance of lubricant monitoring is becoming more apparent with increasing engine performance requirements, increasing engine and maintenance costs, and continuous striving for improved utilization and reliability of equipment.

Currently, the analysis of used lubricants from military turbine engines is being accomplished by the Spectrometric Oil Analysis

Program (SOAP) of the various services. Spectrometric oil analysis determines the concentration of specific wear metals in the oil. The presence of unusual concentrations or the rate at which wear metal concentrations increase indicates abnormal wear conditions.

Although Spectrometric Oil Analysis Programs have been successful and cost effective, spectrometric oil analysis has several drawbacks and limitations. This technique does not provide information on the nature of the abnormal wear; is discriminatory against larger type wear particles which are of prime importance in failure modes; and in many cases, does not identify the specific location of the abnormal wear occurring in the engine.

More recently, a new technique called Ferrography (1) has been developed for the analysis of wear particles. This technique provides information relative to the characteristics of wear particles present and the ratio of the number of large to small particles. The characteristics of the wear particles are categorized as (provide information relative to the wear process such as) spalling, fatique, cutting, corrosion, etc. The amount of particles and the ratio of large to small particles are indicative of the severity of the wear process.

Ferrography is considered a supplementary technique to Spectrometric Oil Analysis in that it provides information not obtainable from SOAP, aids in the evaluation of SOAP data, and can be more

specific in identifying the type and location of the abnormal wearing component.

Having determined the wear particle characteristics and the quantity of various wear particle sizes using ferrography, the question arises as to the composition of the various type wear particles. Scanning electron microscopy (2) has been used successfully for the analysis of some wear particles but has limited application in routine monitoring due to analysis time, cost, and individual particle analysis capability. Information required is not typically the composition of a specific particle, but how many particles are present having this composition. This analytical capability for routine monitoring requires relatively fast analysis time, low equipment cost, technique simplicity, and specificity in the identification of alloys as well as specific metals.

2. Program Objective and Scope

The objective of this program is to develop a systematic method for the identification of wear particles. This objective is being pursued through coupling of microchemical methods with ferrography. Basic approach involves a) investigation of known chemical reactions for identifying specific metals, b) identification of those reactions which have microchemical/light microscopy capability for identifying small particles, and c) development of a

systematic procedure for identification of wear particles, preferably without being removed from the ferrogram slide.

Techniques for identifying wear particles by pre-treating the lubricant samples containing the wear particles prior to the ferrographic analysis are also being investigated.

This phase report describes the work conducted to date in the area of developing a ferrogram reaction chamber, investigation of potential chemical reactions, and the development of micro-chemical techniques for specific wear particles.

SECTION II

WEAR PARTICLE ANALYSIS CONCEPT

1. Ferrographic Analysis Technique (Ferrogram)

Ferrography is a technique by which wear debris and contaminant particles are separated from a lubricant and analyzed by light microscopy and densitometer measurements. The ferrograph (Figure 1) consists basically of a magnet designed to develop a field having an extremely high gradient near the poles, a pump to deliver the oil sample, and a treated transparent slide on which the particles are deposited.

The oil sample, diluted with a solvent to promote the precipitation of wear particles, is pumped across a transparent slide which is mounted at a slight incline over the magnet. Because the distance from the surface of the slide to the pole faces is slightly greater at the oil delivery than at the oil exit end, the magnetic field gradient is weaker at the delivery than at the exit end, which results in the particles being subjected to a continually increasing magnetic force as they flow along the slide. Particles having different magnetic susceptibility and different size are deposited at different locations with the larger particles normally being deposited at the delivery end of the ferrogram slide and the smaller particles being deposited further down the slide. After the oil sample has been pumped over the slide, the slide is washed with a solvent to remove the residual oil and to "fix" the particles to the slide.

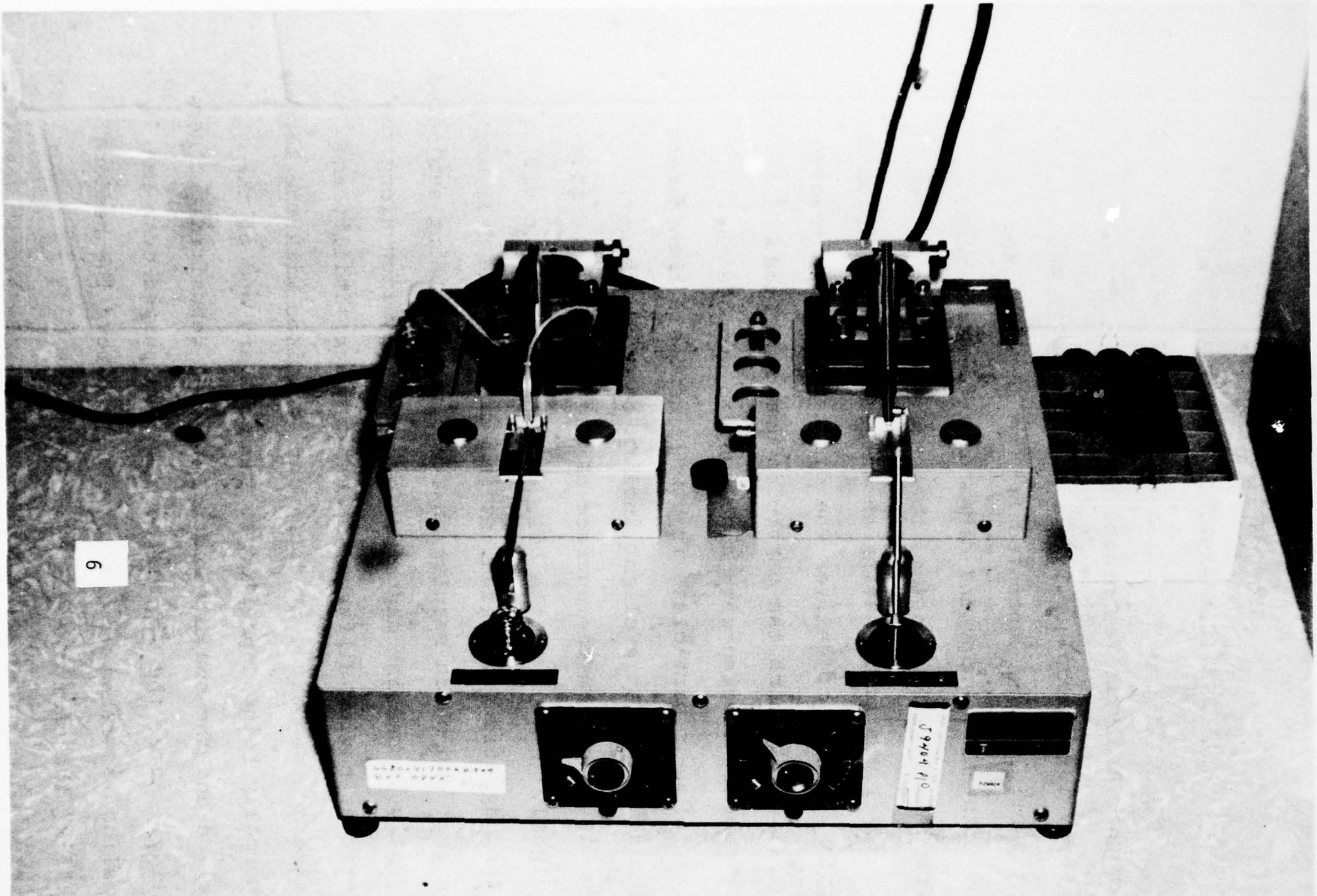

FIGURE 1. FERROGRAPH

The quantity of deposited particles is then determined by making densitometer measurements at specified locations along the slide. The current technique is to make the densitometer measurements during the microscopic examination of the particles (2). The amount of deposit at a specific slide location is reported in percent area covered. Readings are normally taken at the region of maximum coverage near the delivery point of oil onto the ferrogram (approximately 54 mm from the exit of the ferrogram) and at a point 50 mm from the exit of the ferrogram. These density readings have been denoted A_L and A_S respectively. The parameter S_A (Severity of Wear Index) is calculated according to the equation

$$S_A = A_L^{\,2} - A_S^{\,2} \qquad \text{Equation 1}$$

This parameter has been found to be sensitive to the onset of severe wear. The general level of wear is normally indicated by the quantity $A_L + A_S$ and is used to detect certain noncatastrophic wear modes such as excessive rubbing wear and some types of oxidative wear. The severity index is used to indicate the onset of a severe wear mode that may quickly result in failure.

The morphology of particles collected on the ferrogram slide is determined by microscopic examination using either or both transmitted or reflected lighting. Bichromatic lighting (green transmitted and red reflected) is also used and aids in particle identification. Polarized light is also being investigated for distinguishing specific type particles. Figure 2 is a photograph of a Reichert Zetapan microscope being used for these studies. Particle charac-

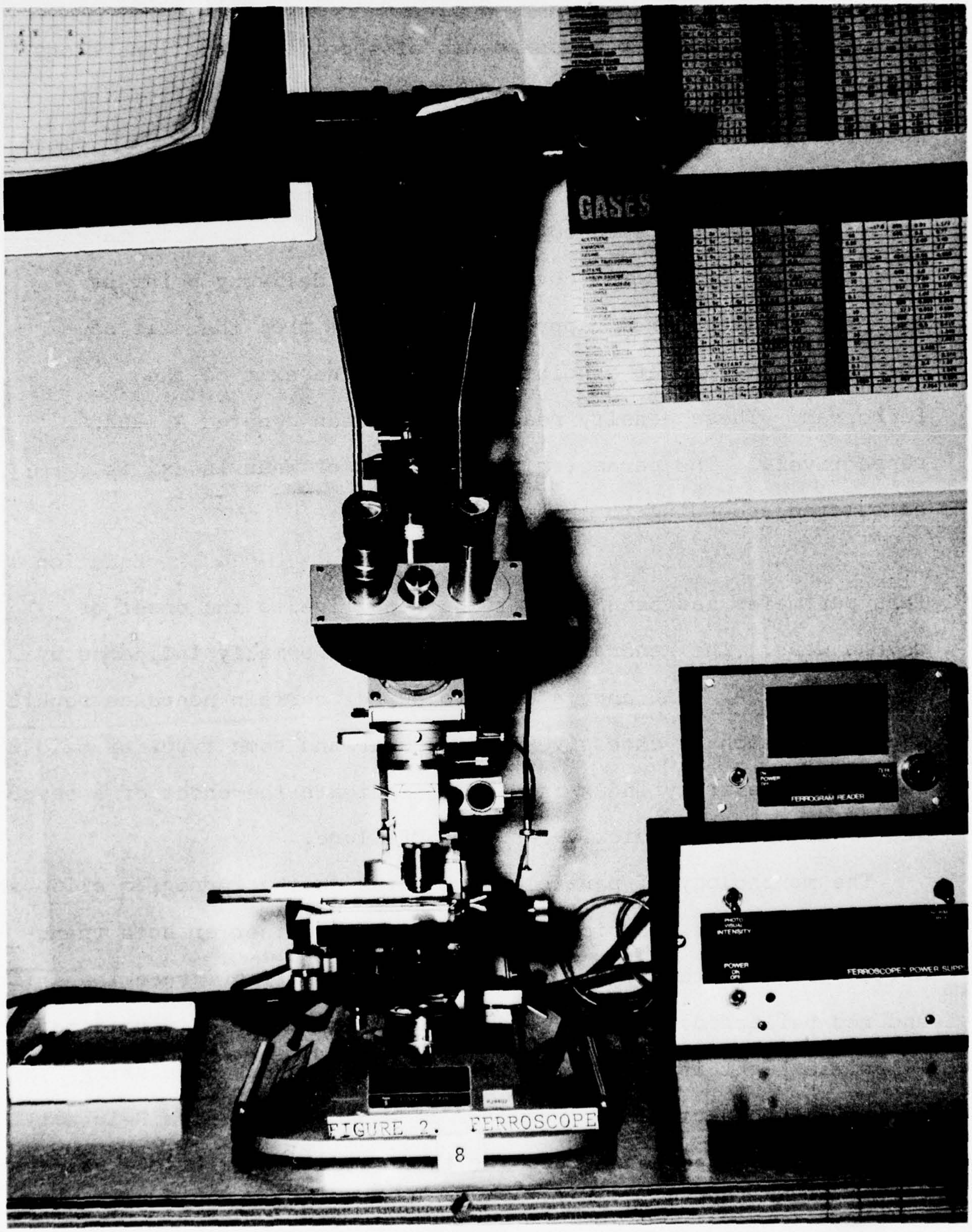

FIGURE 2. FERROSCOPE

teristics obtained from the microscopic examinations include size, shape, surface features, color, degree of magnetic susceptibility, and transparency.

2. Chemical Microscopy

The term chemical microscopy was coined many years ago (3) for the purpose of differentiating between microchemical methods and the application of the microscope to the solution of chemical problems. Originally the term microchemical methods referred to qualitative analyses observed using microscopy but later included many techniques developed to deal with minute quantities of materials without use of the microscope.

In this study chemical microscopy refers to the identification of wear particles and contamination debris using microscopy after the specific particles have been chemically tagged either prior to or during microscopical observations or after selective removal of specific type particles.

Chemical methods being investigated include oxidative and chemical staining, crystal formation on particle surface, selective solution, solution and deposition, and etching.

3. Coupling of Ferrography and Chemical Microscopy

The objective of this program, to provide the capability of identifying all particles of a particular composition after being collected on the ferrograph slide, levies additional requirements

over standard chemical microscopy techniques. Quantitation is required, not in terms of concentration (ppm) of a specific element, but in the number of particles having similar composition. This requirement is being investigated through the following approaches:

a. Pre-Treatment of Particles:

Using this approach, the particles are treated prior to ferrographic separation. This includes tagging the specific particles without effecting their removal by ferrography or by dissolving or removing specific (type) particles. In the case of removing the particles, two ferrograms on the sample would be required, one on the pretreated sample and one after sample treatment.

b. Post-Treatment of Ferrograms:

Using this approach, the particles are treated and identified after being collected on the ferrogram.

The preferred approach is direct post-treatment of the ferrograms. However, in some cases this may be difficult such as the task of distinguishing between different oxides.

SECTION III

EXPERIMENTAL EQUIPMENT

1. Ferrographic Reaction Chamber

A ferrographic reaction chamber was designed and constructed for treating wear particles by various techniques under controlled environmental conditions (Figure 3). The design and construction of the chamber was conducted prior to this effort. However, since the chamber is being used for this program, the checkout and modification, as required, was conducted under this program.

The ferrograph reaction chamber permits mounting of a prepared ferrogram, removing the fixer without removing the magnetic wear debris, and treating the wear debris by gaseous or liquid reagents. Controlled heating of the ferrogram to known temperatures can also be accomplished. The unit consists primarily of a magnet platform, quartz heater, thermocouple and temperature readout and control system, heater hood, gas hood, and two buret systems. A drawing of the reaction chamber (Figure 4) and a parts list (Table 1) are given.

The evaluation and checkout of the reaction chamber revealed the following deficiencies:

a. Thermocouple placement: The original design placed the thermocouple in a well near to and under the wear debris track of the ferrogram. Due to the rapid heat-up and "line" concentration of heat of the quartz heater the temperature measured by the thermo-

A. Reactant pipet

B. Ferrogram heater

C. Digital temperature indicator

D. Gas hood

E. Ferrogram mount

F. Controls

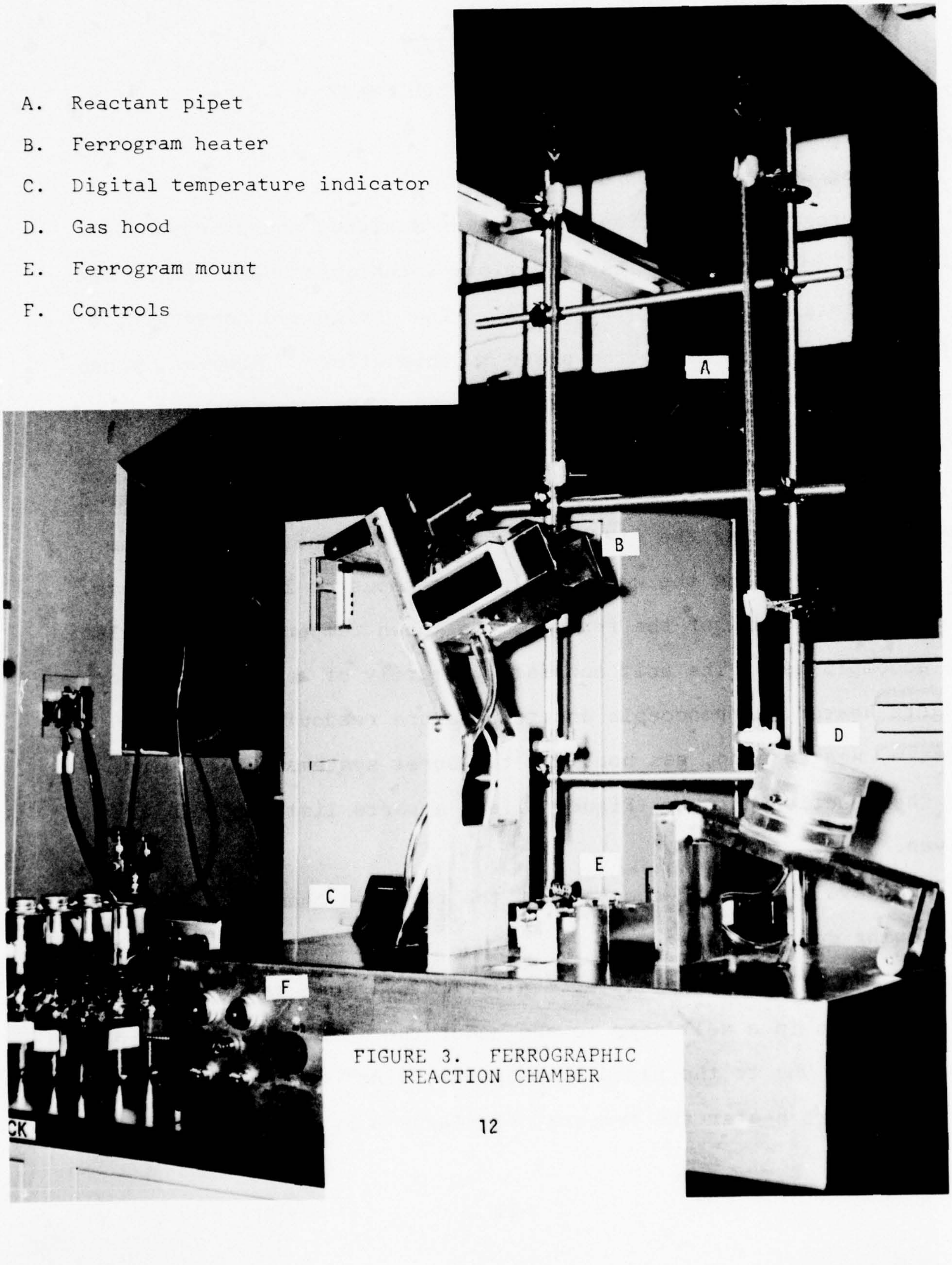

FIGURE 3. FERROGRAPHIC REACTION CHAMBER

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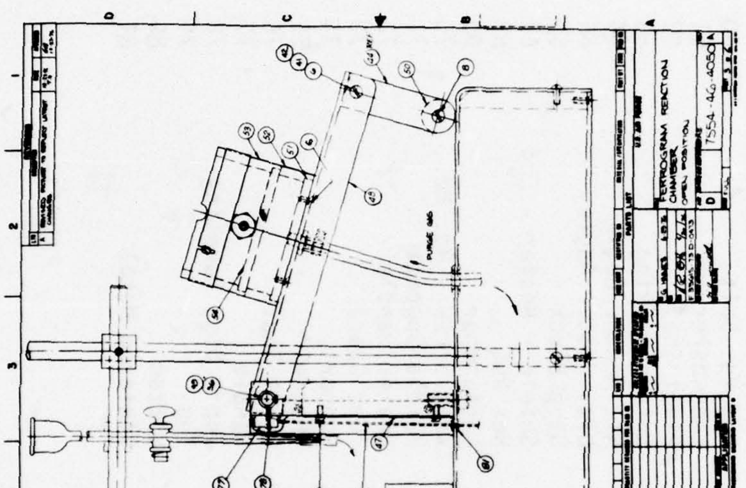

FIGURE 4. REACTION CHAMBER DRAWING

TABLE 1. REACTION CHAMBER COMPONENTS

Item No.	Description	Item No.	Description	Item No.	Description
1	Deck - Chassis	28	Tube	55	Middle Support
2	Bottom	29	Rt. Delivery Tube	56	Cover Tube Assembly
3	Center Support	30	Lft. Delivery Tube	57	Swivel Post
4	Rt. End Support	31	Shield - Heat Sides	58	Set Screw
5	Pan Hd. Screw	32	Pivot Block - Heater	59	Pan Hd. Screw
6	Flat Hd. Screw	33	Hex Hd. Bolt	60	Split Lock Washer
7	Lft. End Support	34	Lockwasher	61	Pan Hd. Screw
8	Meter Housing	35	Pivot Shaft	62	Flanged Inlet
9	Sheet Metal Screw	36	Retaining Ring	63	Relay
10	Nylo-Seal Tubing	37	Rt. Heater Angle	64	Toggle Switch
11	Hose Clamps	38	Lft. Heater Angle	65	Lamp Holder
12	Hose Connector	39	Stop Block	66	T/C Transmitter
13	Line Heater	40	Shield - Heater - End	67	Pan Hd. Screw
14	Grommet	41	Hex Nut	68	Digital Panel Meter
15	Panhead Machine Screw	42	Lockwasher	69	Dimmer
16	Inlet Water Fixture	43	Wood Screw - Rd. Hd.	70	Oval Hd. Screw
17	Hose Connector	44	Handle Support	71	Cable
18	Gas Fixture	45	Heater Handle	72	Lamp
19	Hose Connector	46	Tube Guard	73	Socket
20	Hose Connector	47	Support	74	Clip
21	Microburet - 5ml.	48	Channel	75	Socket
22	Vertical Support	49	Pivot Shaft	76	Switch Actuator
23	Support Clamp	50	Handle	77	Switch, Leaf Actuated
24	Horizontal Support	51	Top - Disc Mounting	78	Pan Hd. Screw
25	Buret Clamp	52	Top	79	Split Lock Washer
26	Pedestal Assembly	53	Bottom	80	Cover, Heat. Safe. Sw.
27	Hex. Hd. Bolt	54	Sintered Metal	81	Grommet

couple was much lower than the actual ferrogram temperature. The new thermocouple design features a bare iron constantan bead mounted over the ferrogram and in the focal line of the quartz heater.

b. Heater and ferrogram alignment: The supports for the heater and gas hoods were not sufficiently rigid to prevent heater and ferrogram misalignment to occur. This misalignment caused poor repeatability in heating of ferrograms. Two locking plates were added to the heater hood to ensure correct repeated positioning of the heater. The ferrogram support block was modified with three screw adjustments for positioning the block under the focal line of the quartz heater.

c. Heater fuse: The 5 amp heater fuse was found to be inadequate and was replaced with a "slo-blow" 7.5 amp fuse.

2. Magnetic Field Strength of Reaction Chamber

A permanent magnet is used for retaining the magnetic wear debris during removing of the "fixer" and subsequent treating of the particles. Since temperatures up to 700°F will be used for identification techniques, possible deterioration of the magnet's field strength must be considered.

The magnetic field strength of the ferrograph reaction chamber and the ferrograph analyzer was determined by using a Bell Model 120 gaussmeter.

The field strength was determined for four equidistant sites along the exposed pole of the reaction chamber magnet. These values

were 1.4, 1.2, 1.3, and 1.3 kilogauss with the first value being for the oil delivery position and the last value being for the oil exit position.

The ferrograph analyzer being used is a dual instrument permitting two ferrograms to be prepared simultaneously. Both magnets were evaluated at positions equivalent to the positions measured on the chamber magnet. Field strength measurements for both poles of each magnet are given in Table 2.

TABLE 2. MAGNETIC FIELD STRENGTH (KILOGAUSS)

	RIGHT HAND UNIT POLE 1	RIGHT HAND UNIT POLE 2	LEFT HAND UNIT POLE 1	LEFT HAND UNIT POLE 2
Position 1 (Entry)	1.9	1.9	1.3	1.5
Position 2	1.7	1.9	1.4	1.6
Position 3	1.7	1.8	1.2	1.5
Position 4 (Exit)	1.6	1.7	1.4	1.7

The above data show the analyzer magnets to be slightly stronger than the reaction chamber magnet. As the program continues, the field strength of the reaction chamber magnet will be monitored to identify field strength deterioration due to heating.

3. Ferroscope Densitometer Linearity

To determine the percent area covered on ferrograms generated in portions of this program, and to determine wear severity indexes, it was necessary to use the ferroscope densitometer. This device employs a photocell in the optical path to determine the

reflected energy returned from the ferrogram. The densitometer is used in the incident light mode only to obtain "percent area covered" values from 0 to 100% for debris on ferrograms. However, a procedure to determine the linearity of the scale from 0 to 100% covered was not available. It was theorized that neutral density filters might be inserted into optical path to determine linearity for reference purposes.

Based upon that concept, data were generated using standard Foxboro/Trans-Sonics, Inc., instructions for the Reichert microscope equipped with an 11x objective. The source intensity was reduced by introducing Kodak gelatin filters (0.1 and 0.3 optical density [OD]) and precision glass mounted filters (0.7 and 1.0 OD) into the optical path at the microscope base where the incident beam is reflected upwards prior to incident illumination of the ferrogram. This location was selected due to the convenience of placing the various neutral density filters into the optical path.

Before each percent area covered value was obtained on a ferrogram employing the indicated filter, the densitometer limits were established at 0 and 100%. Base data consisted of five experimental values of percent area covered for each neutral density filter. Fractional area covered values were calculated using the following relationship:

$$\text{Optical density or absorbance} = \log\left(\frac{1}{\text{T, transmittance}}\right)$$

Equation 2

and assuming that reflected densitometer (R) values were related to transmittance as follows:

$$R = 1 - T \qquad \text{Equation 3}$$

and

$$(100)\,(R) = \%\ \text{area covered} \qquad \text{Equation 4}$$

The data generated using neutral density filters are given in Table 3.

The data are very satisfactory, in that experimental values agree quite closely with those calculated. The data indicate that the procedure and equipment used in the experiment are adequate to determine the relative amounts of comparable debris deposited upon ferrograms. The procedure suggested here is useful as a method to determine the linearity of the densitometers for values of interest between 0 and 100% area covered. It should be noted that the basic absorbance of the gelatin and glass filters were not considered, because those values were thought to be minor compared to optical densities of 0.1, 0.3, 0.7, and 1.0.

TABLE 3. DENSITOMETER LINEARITY DATA

Filter Optical Density (± 5%)	T(calc.)	R(calc.)	R(found)	Range	(σ)	Found %Area Covered
0.1*	0.795	0.205	0.193	0.191-.194	±.016	19.3
0.3*	0.501	0.499	0.479	0.476-.481	±.021	47.9
0.7**	0.200	0.800	0.821	0.819-.824	±.018	82.1
1.0**	0.100	0.900	0.913	0.911-.914	±.013	91.3

* Gelatin neutral density filters (Eastman Kodak, Rochester, N.Y.)

** Precision neutral density filters (Melles Griot type)

T = Filter Transmittance

R = Reflected densitometer value

σ = Standard deviation

SECTION IV

EXPERIMENTAL INVESTIGATIONS

1. General

Several techniques have been investigated for the specific identification of wear particles, including pretreatment of particles prior to ferrography and particle treatment after ferrography. For some investigations, appropriate wear particles were mechanically generated from sheet or bar stock of specification materials. For other studies, reagent grade metal powders were used. These metal particles were blended with Specification MIL-L-7808 aircraft turbine lubricant for preparing required ferrograms. Metals thus far used for this study are given in Table 4.

TABLE 4. METALS USED FOR IDENTIFICATION STUDIES

METAL	SPECIFICATION
Copper	QQ-C-576
Cast Iron	QQ-I-625
Steel, Carbon, Grade 1009	QQ-S-698
Silicon Bronze	AMS-461B
Waspaloy	AMS-5544
Steel, Tool (Grade M-50)	AMS-6490A
Iron Powder (325 Mesh)	Amax Co.
Aluminum Powder (325 Mesh)	Fisher Chem. Co.

The different approaches investigated are described in the following sections including experimental techniques used and the results of these preliminary investigations.

2. Temperature Coloring

The use of temperature coloring, sometimes referred to as bluing, was investigated for three types of iron alloys. Ferrograms were prepared from MIL-L-7808 lubricant samples containing suspended particles (metal filings) of the three iron alloys. Each ferrogram was then heated at various temperatures for 90 seconds. Heating of the ferrograms was done on a hot plate with surface temperatures being measured by use of a previously calibrated surface thermometer. Results from this investigation are given in Table 5.

Concurrently with this effort, Barwell et al. (4) reported on investigating temper colors in ferrography for various iron alloys not studied in this effort. Similar temperature coloring was obtained for similar alloys for both programs.

Future efforts in this area will include investigating other type alloys, mixtures of alloys, and temperature coloring environment.

3. Chemical Bluing

One concept that offers potential in the identification of wear particles on ferrograms is a technique referred to as chemical bluing. The proposed procedure is to use chemical oxidants as used for gun and metal bluing to mildly and selectively oxidize wear metal debris. Texts on bluing and gunsmithing contain several "recipes" and formulas which appear to be potentially useful. For

TABLE 5. TEMPERATURE COLORING OF METAL PARTICLES ON FERROGRAMS

TYPE PARTICLE	PARTICLE APPEARANCE BEFORE HEATING	PARTICLE APPEARANCE AFTER 90 SEC EXPOSURE		
		550°F (287°C)	600°F (315°C)	650°F (343°C)
Tool Steel (M-50)	Bright Metallic Luster	Few (less than 1%) turned blue. Most particles showed no change.	Approximately 75% of particles were blued. Even bluing occurred for all size particles	Most particles were blued. Few large particles remained unchanged.
Carbon Steel (Gr 1009)	Bright Straw Color	Few (less than 1%) turned blue. Most particles showed no changes.	Approximately 75% of particles were blued. Few larger particles yellow-brown in color.	Most particles bluish gray. Few remained yellow-brown.
Cast Iron	Dull Dark Color	No change in particle color or appearance.	Most particles light straw color.	Approximately 2% of the particles were blued and all were 3 to 10 microns in size. Most particles remained straw color.

example, several formulas given by Chapel (5) are readily adaptable for bluing and browning. The traditional bluing processes are divided into classes requiring a) paint and lacquers, b) chemicals without heat, and c) chemicals with heat and water. Of course, the first class is without merit for this program. The second class is ideal for use in this program but methods are not readily found. One common formula offered with variation is one that contains potassium nitrate, potassium chlorate, mercury chloride, nitric acid, and water. This bluing agent requires hot water in several of the suggested bluing steps. Another blue is composed of sodium nitrate, sodium hydroxide,and water. The bluing procedure used with this material also requires rather high solution temperatures. Another formula offered for stainless steels after removing grease and oil is a coppering solution. Containing sulfuric acid, copper sulfate, and water, the solution is used to deposit a light copper coating on the stainless steel. The coating is then treated with nitric acid prior to bluing, using one of the common blues. All of the above bluing formulas and associated methods have potential utility for wear particle identification. However, they have not been evaluated. Modern commercial gun blues may be used at room temperature. At least one of those contains selenium dioxide (6) and the use of selenium dioxide as an oxidizing agent is well documented (7). The possibility of using similar materials for mild room temperature bluing encouraged some limited experiments to determine if the method

had potential. Initially, a saturated solution of SeO_2 in methanol was prepared. When the solution was placed on a soft steel polished surface, the surface browned quickly. The rate at which the commercial preparation attacked the metal surface was found to be more rapid, suggesting that additional agents may have to be incorporated into the solution. A milder solution (0.5 g SeO_2/40 ml methanol) was prepared and pumped over a freshly prepared ferrogram on which low alloy wear debris had been deposited. The experiment was not successful in that no microscopically visible discoloration of the metallic debris occurred under those gentle conditions. However, due to the high potential of a room temperature bluing or coppering method, it is expected that these methods will be pursued. Methods exist to increase the activity of the solution and should promote bluing of selected metallic debris.

4. Copper (II) Chloride Dissolution

Aqueous solutions of copper (II) chloride have been used to remove metal foils from cast polymeric materials. To determine the relative effectiveness of the technique to preferentially remove one metal from a mixture on a ferrogram, e.g., aluminum from steel, and to determine if aqueous solutions could be used for the treatment of ferrograms, a simple experiment was devised.

The experiment consisted of the preparation of ferrogram 4951, using 3 ml of a MIL-L-7808 synthetic turbine lubricant containing 0.5 mg/ml of a 325 mesh aluminum powder and 0.5 mg/ml of a 325 mesh iron powder. After the sample was pumped over the slide, 3 ml of

trichloroethylene was used to remove traces of the oil. The slide was permitted to air dry so that the slide could be inspected microscopically and densitometer values could be determined. The ferrogram was then remounted in the ferrograph and treated with 5 ml of copper (II) chloride solution (10g $CuCl_2$ in 100 ml H_2O). The slide was then allowed to dry followed by fixing, using standard procedures. The slide was again inspected and densitometer values determined. Those data are given in Table 6.

TABLE 6. FERROGRAM 4951 DENSITOMETER DATA

Slide Position (mm)	% Area Covered Pretreatment	% Area Covered Post treatment
Entry	53.3	53.5
50	1.9	6.1
40	3.8	27.7
30	1.4	23.8
20	29.1	30.5
10	2.0	35.4

When the slide was inspected prior to treatment, it was found that the ferrous appearing debris was concentrated near the entry while aluminum appearing debris was found on most of the wear track but heavier near the exit end. It should be restated that the metallic debris was relatively large particles and that there was little tendency for the aluminum to precipitate and mix with the ferrous debris as might be expected from materials generated by Fe/Al contact. However, after treatment it appeared that the ferrous as well as portions of the aluminum debris had been attacked leaving oxide debris present in the original sample along with debris

generated in the treatment. At the concentrations employed and without improving the washing technique, the method does not appear to be attractive.

5. Sample Pretreatment Technique

Infrequently, lubricant samples submitted for ferrographic analysis contain excessive amounts of metallic or non-metallic debris which hinders the analysis of other types of particles present in lesser quantities but being of prime importance. Procedures which include techniques to remove oxides, metals, or a selected metal only from the mixtures have been proposed. Of those methods, one suggested by Klaus, et al. (9) for separating oxides and metallic particles was evaluated in preliminary experiments.

For mixed debris samples, an HCl (4N) and acridine (0.001N) solution is used to attack the oxide debris. For selective attack on metallic debris, a 6N nitric acid solution is employed. Then pretreatment of samples with either of the above reactants to eliminate the metallic or non-metallic debris results in a sample that is more easily analyzed.

To evaluate the technique, Fe_2O_3 particles, 1009 steel filings or M-50 steel filings were individually added to synthetic turbine lubricant so that concentrations in each of the reference samples was 1 mg debris/ml of oil.

Five ml of the oxide reference sample was treated with an equal volume of the HCl/acridine solution. After allowing the phases to stand and separate, a ferrogram was prepared from the lubricant layer and compared to a ferrogram of the referenced oxide sample. Based upon densitometer readings and microscopic examinations of the slide, the treatment removed nearly all the oxide. However, the effect of debris settling was not considered in this initial test and probably was responsible for some loss of oxide.

Similarly, a prepared sample of 1009 steel filings in synthetic turbine lubricant was treated with 6N nitric acid. Ferrograms of the treated and untreated sample indicated the elimination of practically all metallic debris. However, again the procedure used ignored the effects of settling.

To evaluate the utility of the procedure for mixed samples, equal volumes of iron oxide and M-50 steel samples were combined. The mixture was then treated with HCl/acridine solution. A ferrogram was prepared from the lubricant layer. Microscopic examination of the ferrogram revealed that much of the oxide debris was no longer present while some M-50 debris remained. Some oxide was noted which was of slightly different color than that present in the reference sample.

The reference sample exhibited about equal portions of oxide and M-50 debris along the slide with the oxides more concentrated near the exit end of the ferrogram.

Another mixed oxide/M-50 sample was treated with HNO_3 to determine if selective attack on the metallic debris was possible. A ferrogram of the treated sample showed a moderate concentration of oxides and a few widely scattered metallic particles. The concentration of oxides was not as great as in the reference sample, which may be due to settling losses.

The above experiments indicate that a pretreatment technique does have potential in simplifying ferrographic analysis of mixed debris samples. However, additional efforts will be necessary to determine the significance of settling, to further examine the process, and to optimize variables as required. Also, it was noted that none of the pretreatment procedures were quantitative in selective debris removal; i.e., for the mixed debris sample, both types of debris were present after treatment in each case, but the ratios had been altered significantly.

Similar pretreatment schemes are being considered for the selective removal of one type of metallic debris from other types in mixed samples. Although the potential is high for such methods, development of the techniques remains to be accomplished.

6. Crystal Formation on Particle Surfaces

The organic compound 1,2-dyhydroxycyclobutenedione commonly known as "squaric" acid has been studied by several investigators (9) but relatively little use has been made of the complex forming

ability of the squarate ion. Microcrystalline derivatives are produced when metals are immersed in solutions of squaric acid. Iron (III), uranium (VI), silver (I) and copper (II) ions have been precipitated as characteristic derivatives from aqueous solutions of squaric acid (10).

Preliminary investigation of forming microcrystalline structures on wear particle surfaces when treated with aqueous solutions of squaric acid has produced positive results. In these experiments wear particles generated from soft iron, high alloy iron, and bronze have been investigated. A few particles of each metal were placed on microscope slides and microquantities of 0.02M squaric acid solution were added to the particles. Results of these tests are given in Table 7.

TABLE 7. EFFECT OF SQUARIC ACID ON METAL PARTICLES

	Metal Particle Type			
Test Time (min)	QQ-C-576 Copper	AMS 4616B Bronze	AMS 1009 Steel	AMS 5544 Waspaloy
5	Rosettes of yellow crystals on metal particles	No change	No change	No change
10	Same as above	No change	No change	No change
15	Sane as above	Yellow crystalline spines growing from metal particles	Red coloration on particle surface. Few red crystalline particles were formed.	

Additional work with squaric acid for particle identification will include variation of test temperature, test time, normality of the squaric acid solution, and the effects of squaric acid on other type metal particles.

CONCLUSIONS

This research has shown that coupling of microchemical/ light microscopy techniques with ferrography can provide techniques for the identification of wear particles.

Temperature coloring offers a useful procedure for distinguishing between various ferrous metals. Colors obtained for various iron and steel particles, when heated to specified temperatures, agree with published values for metals investigated. The use of the ferrographic reaction chamber in subsequent effort will enable fuller utilization of this technique. Chemical bluing appears to have equal potential but will require additional development before becoming a satisfactory identification technique.

Crystal formation on wear particle surfaces appears to have good potential for wear particle identification. Additional investigation in this area will include use of other complexing agents, variation of test time and temperature, optimizing normality of squaric acid solutions, and the complexing of squaric acid with other type metals and alloys.

The copper chloride dissolution technique does not appear as promising as other techniques for wear particle identification due to lack of specificity.

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